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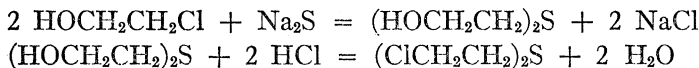
## ETHYLENE CHLORHYDRIN

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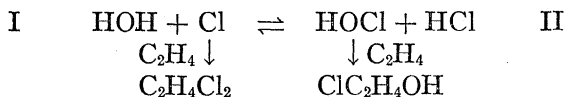
Communicated, July 30, 1919

Within a few days after the so-called "Mustard Gas" was introduced (July 12-13, 1917) as a means of offence, it was definitely identified as  $\beta$ - $\beta$ -dichlorethylsulphide. There was good reason to believe that it had been manufactured from ethylene chlorhydrin, according to the method described some thirty years previously by V. Meyer.<sup>2</sup> The reactions involved in that method are these: (1) Ethylene chlorhydrin in solution reacts with sodium sulphide and gives, in good yield, dihydroxyethylsulphide, which is non-poisonous; (2) this product gives on treatment with concentrated hydrochloric acid the highly toxic  $\beta$ - $\beta$ -dichlorethylsulphide:



The problem however was,—how to get the chlorhydrin itself? From the practical standpoint, a process based upon the additive reaction between ethylene and hypochlorous acid seemed most promising, notwithstanding the facts that this acid could only resist in concentrations of 1 to 3%, and that the best yield of chlorhydrin by this method was known to be not more than 30% of the theory.

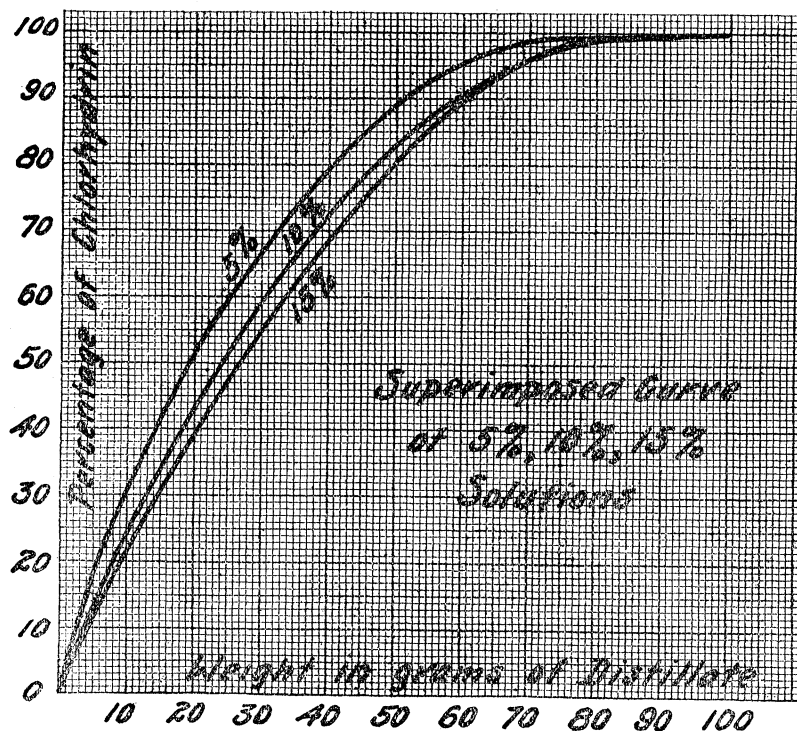
The reaction between chlorine and water was employed as the source of hypochlorous acid. As is well known, this reaction yields extremely little hypochlorous acid, the equilibrium favoring largely the left-hand side of the equation. Nonetheless, it was found that if ethylene and chlorine in equimolecular amounts are passed into cold water and the mixture is well stirred, chlorhydrin, and not ethylene chloride, is the principal product. This is explained on the assumption that reaction II proceeds with considerably greater velocity than reaction I.



While a concentration up to 15% of chlorhydrin can be attained in this way, in practice it has been found advisable to stop with a concentration of 7 to 8%. The progress of the reaction is ascertained by distilling a small sample of the product and determining its refractive index, that of water being 1.333 while that of chlorhydrin was found to be 1.442.

It was established that not only is it unnecessary to keep on neutralizing the hydrochloric acid produced with the progress of the reaction, but that in fact it is inadvisable to do so.

*Separation of chlorhydrin.*—Pure chlorhydrin boils at  $128^{\circ}\text{C}$ . It was found, however, that when mixed with water in proportion of 42.5 parts of the former and 57.5 parts of the latter, the two form a constant boiling mixture which distills at  $95.8^{\circ}$ . Consequently, aqueous solutions of chlorhydrin poorer than 42.5% tend to give on distillation initial frac-



tions approaching in composition the constant boiling mixture; solutions richer than 42.5% give as the final fractions pure chlorhydrin. With the addition of salt or calcium chloride to the liquid to be distilled, concentrations of chlorhydrin up to 80% can be readily obtained. The diagram shows what happens when 5, 10 and 15% solutions of chlorhydrin are subjected to distillation.

Chlorhydrin is miscible with water in all proportions. It has, however, been found that it can be salted out from its aqueous solutions, provided that the proper conditions as to initial concentration, etc., are

observed. Solutions containing 70% of chlorhydrin can readily be obtained in this manner.

It was also found possible to extract the chlorhydrin from its aqueous solutions by some immiscible solvent, benzene suggesting itself as the best from the practical standpoint. Thus, by a judicious combination of the three methods,—distillation, salting out and extraction,—chlorhydrin could readily be obtained in any desired concentration and purity.

<sup>1</sup> The investigation was done under the auspices of the Bureau of Mines, War Gas Investigations Department, December 1917–July, 1918. The paper in detail, approved for publication by Major-General William L. Sibert, Director of the Chemical Warfare Service, U. S. A., will appear in the *Journal of the American Chemical Society*.

<sup>2</sup> Meyer, V., *Berlin, Ber. D. chem. Ges.*, **19**, 1886, (3260), Clarke, H. T., *London, J. Chem. Soc.*, **101**, 1912, (1583).

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## STUDIES OF THE CONSTITUTION OF STEEL

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Researches on steel, considered as a solid solution, were carried on in this laboratory by the late J. W. Longley prior to 1876.<sup>1</sup> Other investigations of iron and steel were carried on in the interval between 1876 and 1891, at which latter time the present writer began a series of researches on the chemical constitution of the carbides of iron. The results of these investigations have gone to show that the carbides found in steel have a much more complex structure than would be indicated by the commonly accepted formula  $\text{Fe}_3\text{C}$ . The idea that the carbides of iron and many other metals can best be studied if the carbides are regarded as metallic substitution products of hydrocarbons was advanced<sup>2</sup> in 1896. In addition to recognizing the complex molecular constitution of the carbides, the assumption has been made that the atomic relations existing between the carbides or other solutes dissolved in iron are essentially the same as those which exist between the molecules of substances in aqueous solution and the water in which they are dissolved. Some further evidence in support of the hypothesis of the unity of mechanism of all solutions without regard as to whether the solvent is solid or liquid, metallic or a non-conductor of electricity, is given in two papers, one of which will be read at the Autumn meeting of the Iron and Steel Institute and the other at a forthcoming meeting of the Faraday Society.

In the first of these it is shown that from twelve samples of steels, including straight carbon steel, high silicon, high phosphorus, manga-